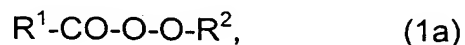


## Patent claims

1. A process for reducing the amount of residual monomers in aqueous polymer dispersions by chemical aftertreatment, wherein the aftertreatment is carried out in the aqueous polymer dispersions with addition of a redox system which comprises

a) from 0.005 to 5% by weight, based on the total weight of all monomers used for the preparation of the polymer dispersion, of at least one oxidizing agent based on an organic peroxide from the class consisting of the

a1) peresters of the general chemical formula



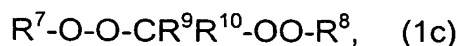
in which  $R^1$  and  $R^2$ , independently of one another, are substituted or unsubstituted alkyl, aryl, cycloalkyl, aralkyl,  $R^3CO$  or  $R^3OCO$ , in which  $R^3$  is unsubstituted or substituted alkyl, aralkyl or aryl, and/or

a2) percarbonates of the general chemical formula



in which  $R^4$  and  $R^5$ , independently of one another, are unsubstituted or substituted alkyl, aryl, cycloalkyl, aralkyl,  $R^6CO$  or  $R^6OCO$ , in which  $R^6$  is unsubstituted or substituted alkyl, aralkyl or aryl, it being possible for the radicals  $R^4$ ,  $R^5$  and  $R^6$  themselves to contain percarbonates, and/or

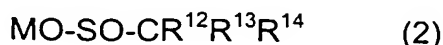
a3) perketals of the general chemical formula



in which  $R^9$  and  $R^{10}$ , independently of one another, are hydrogen or alkyl and  $R^7$  and  $R^8$ , independently of one another, are unsubstituted or substituted alkyl, aryl, cycloalkyl, aralkyl,  $R^{11}CO$  or  $R^{11}OCO$ , in which  $R^{11}$  is unsubstituted or substituted alkyl, aralkyl or aryl,

and

- b) from 0.005 to 5% by weight, based on the total weight of all monomers used for the preparation of the polymer dispersion, of at least one reducing agent from the group consisting of the sulfinic acids and the salts thereof having the structure



in which M is hydrogen,  $\text{NH}_4$ , a monovalent metal ion or one equivalent of a divalent metal ion of the groups Ia, IIa, IIb, IVa or VIIIb of the Periodic Table of the Elements, in which  $\text{R}^{12} = \text{OH}$ ,  $\text{NR}^{15}\text{R}^{16}$ , in which  $\text{R}^{15}$  and  $\text{R}^{16}$ , independently of one another, are hydrogen or  $\text{C}_1\text{-C}_6$ -alkyl, in which  $\text{R}^{13} =$  hydrogen or an alkyl, alkenyl, cycloalkyl or aryl group, it being possible for these groups to have 1, 2 or 3 substituents which, independently of one another, are selected from  $\text{C}_1\text{-C}_6$ -alkyl,  $\text{OH}$ ,  $\text{O-C}_1\text{-C}_6$ -alkyl, halogen and  $\text{CF}_3$ , in which  $\text{R}^{14} = \text{COOM}$ ,  $\text{SO}_3\text{M}$ ,  $\text{COR}^{15}$ ,  $\text{CONR}^{15}\text{R}^{16}$ ,  $\text{COOR}^{15}$ , in which M,  $\text{R}^{15}$  and  $\text{R}^{16}$  have the meanings stated above, or, if  $\text{R}^{13}$  is aryl, this may be unsubstituted or substituted as stated above,  $\text{R}^{14}$  is also H, and the salts thereof.

2. The process as claimed in claim 1, wherein the redox system may additionally contain catalytic amounts of a polyvalent metal ion which may occur in a plurality of valency states.

3. The process as claimed in claim 1, wherein peresters, in particular tert-butyl perbenzoate, tert-butyl peroxy-3,5,5-trimethylhexanoate and/or tert-butyl peroxy-2-ethylhexanoate, particularly preferably tert-butyl perbenzoate, are used as oxidizing agents.

4. The process as claimed in claim 1, wherein percarbonates are used as oxidizing agents, in particular 1-(2-ethylhexanoylperoxy)-1,3-dimethylbutyl peroxy-pivalate, di(2-ethylhexyl)peroxydicarbonate, 2,5-dimethyl-2,5-di(2-ethylhexanoylperoxy)hexane, tert-amylperoxy 2-ethylhexyl carbonate, tert-butylperoxy isopropyl carbonate and/or tert-butylperoxy 2-ethylhexyl carbonate.

5. The process as claimed in claim 1, wherein perketals are used as oxidizing agents, in particular 1,1-di(tert-butylperoxy)-3,3,5-trimethylcyclohexane, 2,2-di(4,4-di(tert-butylperoxy)cyclohexyl)propane, 1,1-di(tert-butylperoxy)cyclohexane, 2,2-di(tert-butylperoxy)butane and/or 3,6,9-triethyl-3,6,9-trimethyl-1,4,7-triperoxonane.

6. The process as claimed in claim 1, wherein 2-hydroxyphenylhydroxymethylsulfonic acid or the sodium salt thereof, 4-methoxyphenylhydroxymethylsulfonic acid or the sodium salt thereof, 2-hydroxy-2-sulfinatoacetic acid or the disodium or zinc salt thereof or 2-hydroxy-2-sulfinatopropionic acid or the disodium salt thereof is used as the reducing agent, preferably the disodium salt of 2-hydroxy-2-sulfinatoacetic acid.

7. The process as claimed in claim 1, wherein the amount of oxidizing agent added is in the range of from 0.02 to 3% by weight, preferably from 0.02 to 2% by weight, particularly preferably from 0.05 to 1% by weight, in particular from 0.05 to 0.5% by weight, based on the total weight of all monomers, and wherein the amount of reducing agent added is in the range of from 0.02 to 3% by weight, preferably from 0.02 to 2% by weight, particularly preferably from 0.05 to 1% by weight, in particular from 0.05 to 0.5% by weight, likewise based on the total weight of all monomers.

8. The process as claimed in claim 1, wherein the reducing agent comprises a mixture of disodium 2-hydroxy-2-sulfinatoacetate in an amount in the range of from 50 to 60% by weight, sodium sulfite in an amount in the range of from 30 to 35% by weight and disodium 2-hydroxy-2-sulfonatoacetate in an amount in the range of from 10 to 15 % by weight, based on the total weight of the mixture.

9. The process as claimed in claim 1, wherein the oxidizing agent and the reducing agent are fed in in succession in separate feeds as components for the aftertreatment of the polymer dispersion.

10. The process as claimed in claim 1, wherein at least one component is fed in by metering.

11. The process as claimed in claim 1, wherein at least one of the components is fed in in portions.

12. The process as claimed in claim 1, wherein first the oxidizing agent and then  
5 the reducing agent are added.

13. The process as claimed in claim 1, wherein the reducing agent is fed in by metering.

10 14. The process as claimed in claim 2, wherein the metal ions for the aftertreatment of the polymer dispersion are added after the oxidizing agent and the reducing agent.

15 15. The process as claimed in claim 2, wherein the metal ions for the aftertreatment are added after the oxidizing agent and together with the reducing agent.

16. The process as claimed in claim 2, wherein the polyvalent metal ions used are iron ions.

20 17. The process as claimed in claim 1, wherein the temperature during the aftertreatment is in the range of from 20 to 100°C, preferably from 25 to 90°C, particularly preferably from 30 to 85°C.

25 18. The process as claimed in claim 1, wherein the aftertreatment is carried out under a pressure in the range of  $\leq 1$  MPa.

19. The process as claimed in claim 1, wherein the aftertreatment is carried out at a pH in the range of from 2 to 9.

30 20. The process as claimed in claim 1, which is carried out using a polymer dispersion having a viscosity in the range of greater than or equal to 100 mPa·s,

preferably greater than or equal to 1 000 mPa·s, particularly preferably greater than or equal to 2 000 mPa·s, in particular greater than or equal to 5 000 mPa·s.

21. The process as claimed in claim 1, which is carried out using a polymer dispersion which contains, as polymerizable monomers, esters of vinyl alcohol and monocarboxylic acids having from 1 to 18 carbon atoms, in particular vinyl acetate, vinyl propionate, vinyl n-butyrate, vinyl laurate, vinyl stearate and/or vinyl versatate, preferably vinyl acetate.

22. The process as claimed in claim 1, which is carried out using a polymer dispersion which contains, as polymerizable monomers, esters of  $\alpha,\beta$ -monoethylenically unsaturated mono- and dicarboxylic acids preferably having from 3 to 6 carbon atoms, in particular acrylic acid, methacrylic acid, maleic acid, fumaric acid and itaconic acid, alkanols having from 1 to 12, preferably from 1 to 8 and in particular from 1 to 4 carbon atoms, in particular methyl, ethyl, n-butyl, isobutyl and 2-ethylhexyl acrylate and methacrylate, dimethyl maleate or di-n-butyl maleate, or nitriles of  $\alpha,\beta$ -monoethylenically unsaturated carboxylic acids, in particular acrylonitrile.

23. The process as claimed in claim 1, which is carried out using a polymer dispersion which contains, as polymerizable monomers, aromatic or aliphatic  $\alpha,\beta$ -unsaturated, unsubstituted or halogen-substituted hydrocarbons, in particular ethene, propene, 1-butene, 2-butene, vinyl chloride, vinylidene chloride, styrene,  $\alpha$ -methylstyrene and/or o-chlorostyrene, ethene and styrene being preferred.

24. The use of a polymer dispersion which has been aftertreated by a process as claimed in claim 1 and which contains a reduced amount of residual monomers, for the preparation of adhesives for wood, paper or plastic, for the preparation of coating materials, such as paints or food coatings, for the preparation of powders or of products for construction chemistry or for the finishing of textiles or paper.

Process for the preparation of aqueous polymer dispersions having very low residual monomer contents and the use thereof

5

The invention relates to a process for reducing the amount of residual monomers in aqueous polymer dispersions by chemical aftertreatment. The aftertreatment is carried out in the aqueous polymer dispersion with addition of a redox system which comprises a) from 0.005 to 5% by weight of an oxidizing agent, which contains an organic peroxide, and b) from 0.005 to 5% by weight of a reducing agent, which contains sulfinic acids or the salts thereof. In addition, the redox system can, if required, also contain catalytic amounts of a polyvalent metal ion which may occur in a plurality of valency states. The aftertreatment can be carried out at a temperature in the range of from 20 to 100°C and at a pH in the range of from 2 to 9. The

10

15

invention also relates to the use of a polymer dispersion aftertreated according to the invention for the preparation of adhesives, coatings, powders or products for construction chemistry or for the finishing of textiles or paper.